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by Robert J. Buzzard and Frank L. Gill

Lewis Research Center

Cleveland, Ohio

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HIGH-TEMPERATURE MECHANICAL PROPERTIES OF A TUNGSTEN - URANIUM DIOXIDE COMPOSITE*

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Lewis Research Center

SUMMARY

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The mechanical properties of a tungsten - 20-volume-percent uranium dioxide composite have been investigated at temperatures ranging from 3000° to 5000° F. The properties of this material appear consistent and are similar to the properties of unalloyed tungsten.

The presence of the uranium dioxide particles improves the short-time tensile strength of tungsten while causing a general decrease in ductility at temperatures above 3000° F. No appreciable difference was observed in the tensile properties of the composite material from tests conducted in both hydrogen and vacuum.

Creep-rupture properties of the composite material were also investigated. The creep resistance of this simulated nuclear fuel material appears to be adequate for certain potential nuclear propulsion systems. Conf. R. D. Author

INTRODUCTION

Interest in a water-moderated nuclear reactor as a propulsion device for space vehicles has resulted in a study of possible refractory metal-base fuel element materials (ref. 1). Because of the extremely high operating temperatures (~4500° F) contemplated for such a reactor, uranium dioxide (melting point, approximately 5000° F) is of prime interest as the fissile material. Tungsten is most favorably considered as the matrix material because of its compatibility with uranium dioxide (ref. 2) and with the propellant hydrogen, as well as its superior mechanical properties at very high temperatures (ref. 3).

Although present design concepts for a water-moderated thermal reactor do not require that the fuel elements serve as structural members, the fuel elements will be subjected to both aerodynamic and thermal stresses in a hydrogen environment. Knowledge of the mechanical properties of the fuel element ma-

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terial is therefore necessary in the design of the reactor. The purpose of this investigation was to evaluate some of the high-temperature mechanical properties of tungsten - uranium dioxide fuel composites in order to provide information needed for conceptual design of a reactor.

Since the proposed reactor is thermal, relatively low fuel loadings (10- to 30-volume-percent uranium dioxide dispersed in a continuous tungsten matrix) are contemplated. In this study of the properties of tungsten - uranium dioxide composites, most of the tests utilized a nominal reference compositon of 20-volume-percent uranium dioxide (tungsten - 20 percent uranium dioxide). The mechanical properties of this material were determined from tensile tests conducted in vacuum and hydrogen between 3000° and 5000° F, and from creep-rupture tests conducted in vacuum between 3000° and 4500° F.

MATERIALS

The tungsten - uranium dioxide composites used in this investigation were prepared by a powder metallurgy technique described in detail in reference 4. Essentially this fabrication technique consisted of blending selected tungsten and uranium dioxide powders, cold compaction in steel dies, and hydrogen sintering at 3200° F. The sintered bars were subsequently hot rolled at approximately 3600° F into thin strips 0.030 inch thick by 1 inch wide by 7 inches long. A final density of approximately 17.3 grams per cubic centimeter, or 98 percent of theoretical density, was consistently achieved for the 20-volume-percent uranium dioxide composites. The strips were clad on the major surfaces with 0.002-inch tungsten sheet during rolling to produce a metallurgical bond between the core and cladding. The edges of the rolled strips were not clad. The necessity for a dense surface cladding arises from the fact that uranium dioxide has a relatively high vapor pressure at high temperatures and thus has a strong tendency to vaporize at temperatures above 4000° F (ref. 5). The potential loss of uranium dioxide from the composites can be effectively minimized by the application of a thin surface layer of tungsten (ref. 6).

Metallographic examination (fig. 1) of the test material after fabrication revealed a structure of slightly elongated uranium dioxide particles in a matrix of recrystallized tungsten. The average tungsten grain diameter measured in the rolling direction was found to be approximately 8×10^{-3} centimeter.

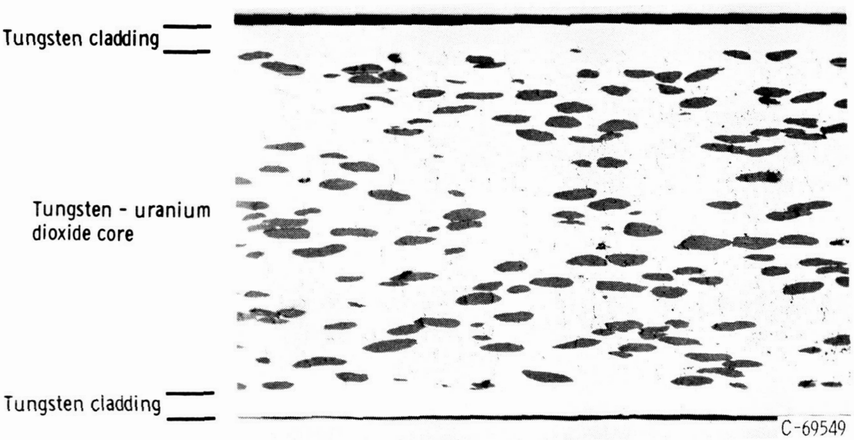

Figure 1. - Microstructure in longitudinal direction of as-rolled tungsten - 20-volume-percent uranium dioxide fuel plate. Unetched. X100. (Reduced 33 percent in printing.)

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APPARATUS AND PROCEDURE

Test Specimens

Tensile and creep-rupture specimens used in this study are illustrated in figure 2. The specimens were machined from the rolled plates by the electrical discharge technique. It is important to note that the tensile axis of the specimens coincides with the rolling direction of the fabricated plates.

To allow for optical measurement of strain during creep tests, 0.020-inch tantalum wires were spot welded to the reduced section of the creep-rupture specimens. The tantalum wires were equally spaced from the center of the specimen with a total gage length of 0.750 inch.

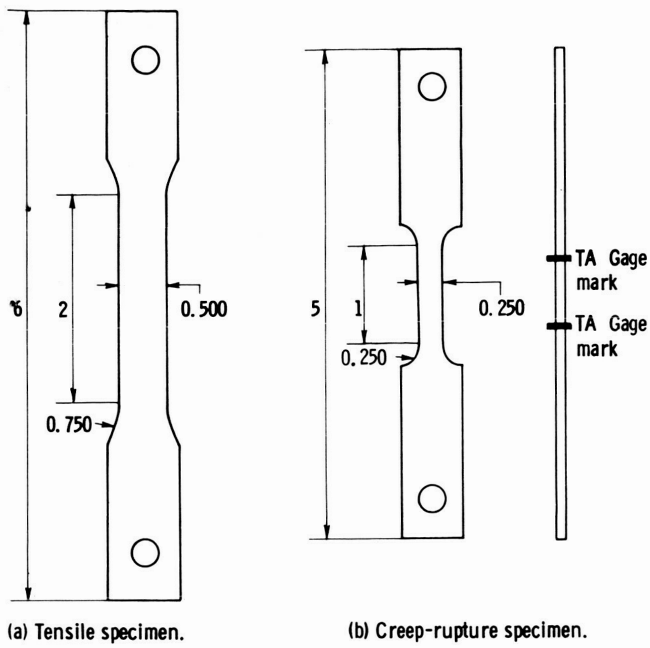

Figure 2. - Test specimens.

High-Temperature Tensile Tests

The majority of the tensile tests were conducted in vacuum (~5×10^{-5} torr) at temperatures ranging from 3000° to 5000° F. Additional tests, however, were conducted in hydrogen (3 psig) to determine the effect of this test environment. A commercial, screw-driven tensile testing machine equipped with a 1000-pound maximum, strain-gage-type, load cell was used for all of these tests. The load cell was connected to a conventional load-time recorder with a standard 12-inch-wide strip chart graduated into hundredths of full scale. Three stations of the recorder were calibrated to record full-scale displacements of 100, 200, and 500 pounds. The vacuum test chamber and the tungsten resistance sheet heater used in this study have been described previously in references 7 and 8, respectively. Specimen temperature was measured with a calibrated tungsten - tungsten-26-percent-rhenium thermocouple wired to the specimen at its midpoint. Measured specimen temperature did not vary by more than ±15° F during any of the tensile tests.

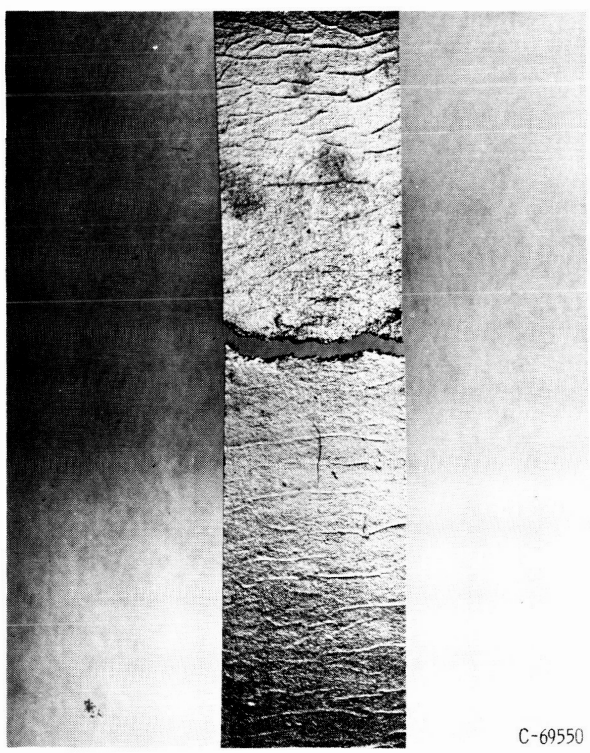

Figure 3. - Typical tensile fracture of composite material.

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Tensile specimens were heated to the desired test temperature in approximately 30 minutes, soaked at temperature for a period of 5 to 10 minutes, and loaded to failure at a crosshead speed of 0.030 inch per minute.

The 0.2-percent yield strength and ultimate tensile strength were determined from the load-time record of the test. Because of the generally jagged nature of the fracture encountered during testing (fig. 3), final elongation measurements were difficult to obtain, and the results were inconsistent. When the final thickness and width of the fractured surface were measured with point micrometers, however, reproducible values for the reduction in area were obtained.

Creep-Rupture Tests

Creep and stress-rupture properties were determined in vacuum ($\sim 5\times 10^{-5}$ torr) on a conventional, constant-load machine at temperatures ranging from 3000° to 4500° F. The test chamber, vacuum system, heating elements, and temperature recording technique were similar to those used for the tensile tests. The creep specimens were heated to the desired temperature and allowed to soak at temperature for 5 minutes prior to the application of load. Loading was accomplished by suspending weights from the specimen within the evacuated chamber. Temperature variation during creep tests did not exceed ±20° F. Specimen temperature was recorded on a strip chart during the test.

Rupture life was obtained from an electric timer designed to stop at the instant of fracture. Creep extension was measured with an optical cathetometer sighted on tantalum wires spot welded to the edge of the specimen. Although the cathetometer was graduated in 0.0001-inch divisions, the gage length readings were reproducible only to within ±0.0002 inch. Creep curves were constructed from the elongation data, and the second stage (steady-state) creep rate was determined from these creep curves.

TABLE I. - TENSILE PROPERTIES OF TUNGSTEN - 20 PERCENT URANIUM DIOXIDE

[2 Mil tungsten cladding.]

Test atmosphere	Test temperature, °F	0.2-Percent yield strength, psi	Ultimate tensile strength, psi	Area reduction, percent
Vacuum	3000	20,000	20,500	28.9
	3000	15,150	16,510	23.0
	3000	16,100	17,750	41.5
Hydrogen	3000	18,650	19,500	14.4
	3000	14,350	15,900	39.5
	3080	21,000	22,800	9.7
Vacuum	3500	8,420	9,290	25.0
	3500	12,600	12,800	(a)
	3500	12,180	12,620	14.3
Hydrogen	3500	11,300	11,900	13.4
	3500	9,600	10,650	13.8
	3500	10,680	11,500	10.2
Vacuum	4000	5,870	6,280	17.3
	4000	5,220	5,930	9.65
	4000	5,480	5,940	(a)
Hydrogen	4000	7,050	8,900	9.8
	4000	6,000	7,700	8.8
	3950	7,120	7,790	---
	4250	5,410	5,915	8.2
	4350	5,180	5,550	8.5
Vacuum	4500	3,245	3,430	9.3
	4500	4,380	4,410	8.5
	4500	3,700	3,940	13.1
	4500	3,610	4,090	9.1
	4750	2,450	3,020	(a)
	5010	1,500	1,530	(a)

[a]Fracture surfaces too irregular to measure accurately.

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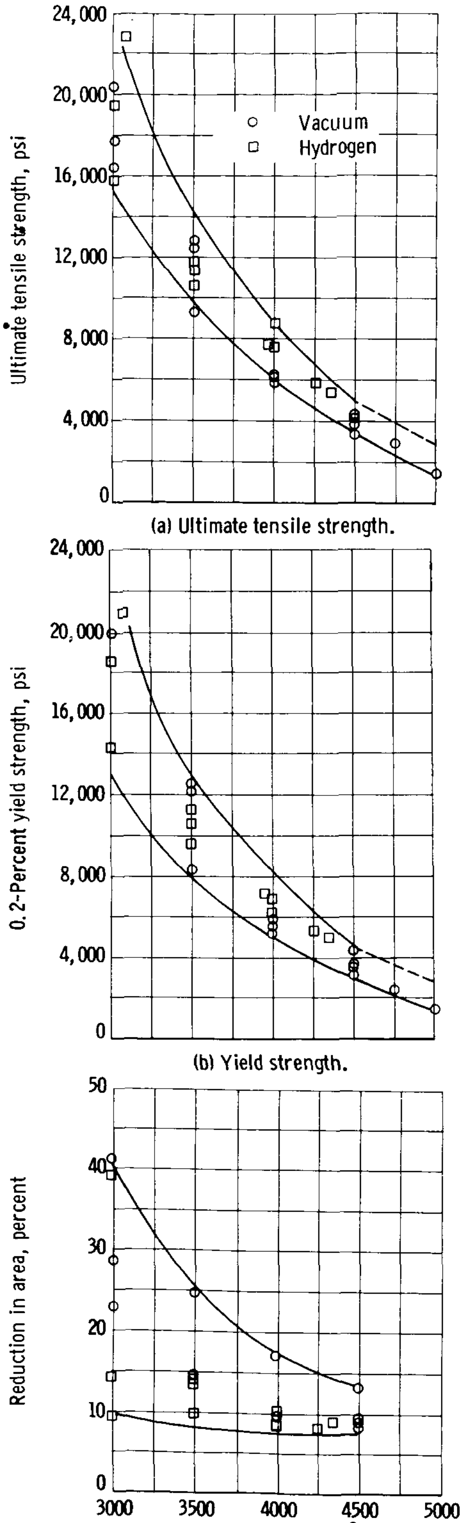

Figure 4. - Effect of temperature on tensile properties of tungsten - 20 percent uranium dioxide. Clad with 2 mil thick coating of tungsten.

RESULTS AND DISCUSSION

Tensile Properties

The results of the short-time tensile tests are given in table I and are shown in figure 4. The average ultimate and 0.2-percent yield strength for tungsten - 20 percent uranium dioxide decreased from approximately 20,000 and 17,000 pounds per square inch, respectively, at 3000° F, to 1520 and 1500 pounds per square inch, respectively, at 5000° F. Tensile ductility, as measured by the reduction in area, decreased from between 9 and 40 percent at 3000° F to between 8 and 16 percent at 4500° F. At temperatures above 4500° F, the fracture was too irregular to permit any reduced area measurements to be made.

A comparison of the average yield and ultimate strength properties of tungsten - 20 percent uranium dioxide with the reported properties of commercially produced, powder metallurgy tungsten sheet (ref. 8) indicates that the tungsten - 20-percent uranium dioxide material is slightly (approximately 5 percent) stronger than commercial tungsten sheet over the temperature range investigated.

To ascertain whether this mild strengthening was due to the presence of the dispersed uranium dioxide particles in the composite or to processing variables, unalloyed tungsten bars and tungsten - uranium dioxide bars of various compositions (10- to 50-volume-percent uranium dioxide) were prepared in an identical manner and tested in vacuum at 4500° F. The results of these tests are given in table II and the average properties are shown in figure 5. From figure 5, it is apparent that additions of uranium dioxide up to 30 volume percent slightly improve the 4500° F tensile strength of tungsten. Since the proposed reactor will utilize fuel loadings of 30-volume-percent uranium dioxide or less, the increased strength of the composite material relative to similarly produced unalloyed tungsten will be useful in the reactor design.

As can be seen from figure 4(c), the tensile ductility of tungsten - 20 percent uranium dioxide is quite inconsistent, particularly at the lower test temperatures (~3000° F). Compared with unalloyed tungsten, the tensile ductility of the ref-

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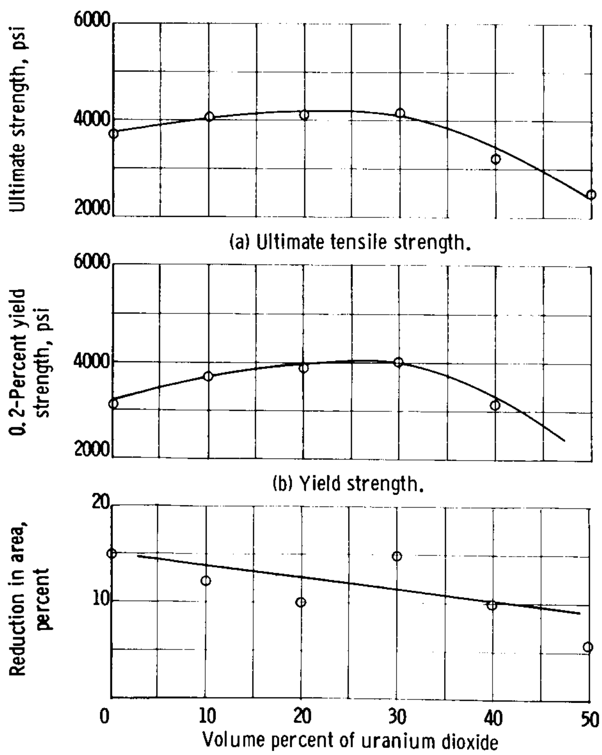
TABLE II. - TENSILE PROPERTIES OF TUNGSTEN-URANIUM

DIOXIDE COMPOSITES AT 4500° F IN VACUUM

[2 Mil tungsten cladding.]

Volume percent of uranium dioxide	0.2-Percent yield strength, psi	Ultimate tensile strength, psi	Area reduction, percent
0	2970	3480	14.1
	2860	3580	15.2
	3650	4160	(a)
10	3590	3950	14.8
	4400	4420	11.7
	3590	3790	10.3
20	4380	4410	8.5
	3700	3940	13.1
	3610	4090	9.1
30	4150	4230	9.4
	3910	4090	18.7
	4050	4270	17.6
40	3145	3225	10.6
	----	3260	9.2
50	----	2550	6.0

[a]Fracture surface too irregular to measure ductility.



(a) Ultimate tensile strength.

(b) Yield strength.

(c) Ductility.

Figure 5. - Effect of fuel loading on 4500° F tensile properties of tungsten-uranium dioxide composites. Clad with 2 mil thick coating of tungsten.

erence composite is generally lower. This lowering of tensile ductility is attributed to the coarse grain structure of the matrix and to the strain concentrating effect of the second phase, as discussed in reference 9, wherein the tensile ductility of composite materials is shown to decrease steadily as the volume percent of the dispersoid increases.

Of more concern than the rather low ductility of the tungsten - 20-percent uranium dioxide composite is the extreme scatter observed at the lowest test temperature (3000° F). Metallographic examination (see photomicrographs of fig. 6) revealed that, in those specimens that exhibited relatively high tensile ductility (>25-percent reduction in area), both the tungsten grains and the uranium dioxide particles near the fracture surface underwent considerable plastic deformation prior to fracture (fig. 6(a)). The fracture in these instances was transgranular, which indicates that the deformation was accomplished by a slip-type mechanism. Further examination revealed that in those specimens that exhibited low tensile ductility (<25-percent reduction in area), fracture occurred by intergranular separation of the tungsten grains without distortion of the uranium dioxide particles (see fig. 6(b)). Since all the tensile specimens tested at temperatures above 3000° F exhibited intergranular fracture, it appears that a transition in the mode of deformation occurs at approximately 3000° F and that the high-temperature deformation mechanism is not as conducive to high ductility as is the low-temperature slip mechanism. Similar results for unalloyed tungsten are reported in reference 10, where high-

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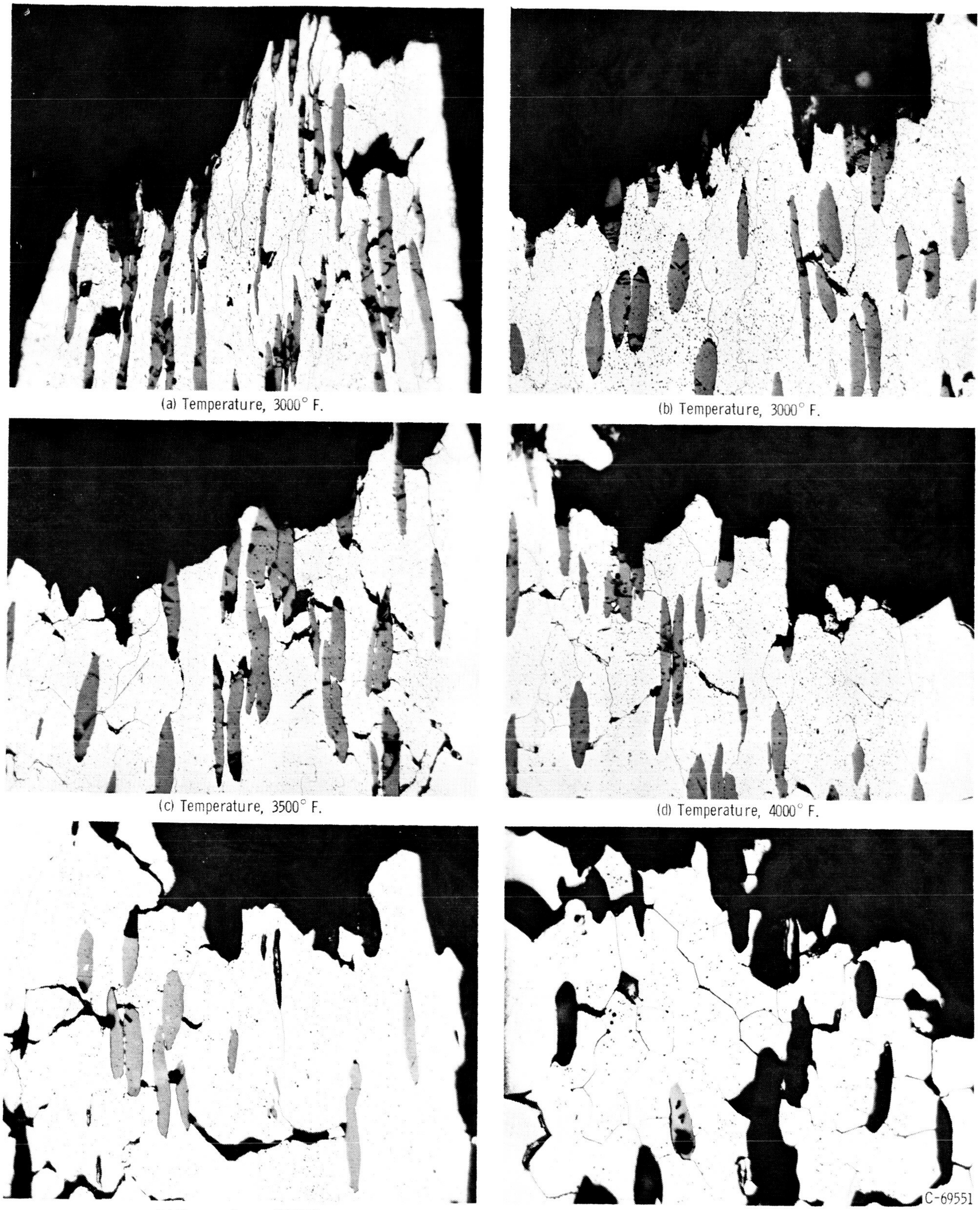

(a) Temperature, 3000° F.

(b) Temperature, 3000° F.

(c) Temperature, 3500° F.

(d) Temperature, 4000° F.

(e) Temperature, 4500° F.

(f) Temperature, 5000° F.

Figure 6. - Representative microstructures near fracture surface of tensile specimens. Etchant, Murakami's reagent; X250. (Reduced 33 percent in printing.)

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temperature ductility is shown to be sensitive to strain rate.

In general, the tensile properties of the tungsten - 20-percent uranium dioxide material tested in hydrogen were similar to the tensile properties observed in vacuum. Although the yield and the ultimate strength of the composite appear to be slightly higher and the ductility lower at temperatures of 4000° F but higher when tested in hydrogen as opposed to vacuum, there is a considerable amount of overlap in the data. Metallographic examination of tensile specimens tested in hydrogen did not reveal any characteristics not observed in vacuum tested specimens.

At temperatures of 4500° F and higher, vaporization of the uranium dioxide particles in the immediate vicinity of the fracture surface was observed (figs. 6(e) and (f)). Since vaporization of the uranium dioxide particles was not observed to occur in regions of the tensile specimen away from the fracture surface, the vaporization is assumed to have occurred during or immediately after fracturing of the specimen.

TABLE III. - CREEP-RUPTURE PROPERTIES OF TUNGSTEN - 20 PERCENT URANIUM DIOXIDE

[2 Mil tungsten cladding.]

Test temperature, °F	Initial stress, psi	Minimum creep rate, in./in./min	Rupture life, min
3000	2220	1.0×10^{-6}	(a)
	2500	7.0×10^{-7}	(a)
	3000	2.0×10^{-6}	(a)
	4000	8.0×10^{-6}	4249
	6000	1.2×10^{-5}	1103
	7000	1.37×10^{-5}	949
	9000	3.8×10^{-5}	257
3500	2220	1.6×10^{-5}	923
	2500	3.75×10^{-5}	527.9
	3000	7.15×10^{-5}	293
	4000	1.3×10^{-4}	165
4000	1500	1.6×10^{-5}	383
	2080	1.13×10^{-4}	117.6
	2200	1.74×10^{-4}	69.1
	2500	1.71×10^{-4}	54.5
	3000	1.66×10^{-4}	37.1
4500	750	6.8×10^{-6}	522.4
	1000	2.15×10^{-5}	112.7
	1500	8.8×10^{-5}	85.4
	2200	(b)	3.7
	2500	(b)	13.4
	3000	(b)	3.1

[a]Test not continued to failure.

[b]Test duration not sufficient for creep rate to be determined.

Creep and Stress-Rupture Data

The data obtained from creep-rupture tests between 3000° and 4500° F are presented in table III. The effect of stress and temperature on the second-stage creep rate of tungsten - 20 percent uranium dioxide is shown in figure 7. For stress levels low enough to permit 10-hour rupture life, the second-stage creep rate of tungsten - 20 percent uranium dioxide increases from approximately 10^{-5} inch per inch per minute at 3000° F to approximately 10^{-4} inch per inch per minute at 4500° F. A comparison of the steady-state creep rate of the

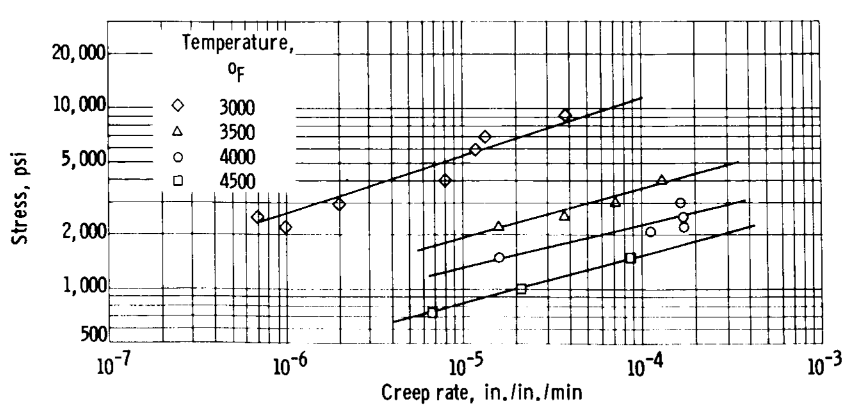

Figure 7. - Minimum creep rate of tungsten - 20 percent uranium dioxide as function of temperature. Clad with 2 mil thick coating of tungsten.

reference material with unalloyed tungsten is difficult because of the wide spread in the reported creep data for unalloyed tungsten between 3000° and 4500° F. For example, comparison of these data with data for sintered and swaged tungsten rods (ref. 11) indicates that unalloyed tungsten is slightly more creep resistant than the composite tungsten - 20-percent uranium dioxide sheet material used in this investigation. Examination of the reported creep rate data for sintered tungsten sheet (ref. 12), however, leads to the conclusion that the tungsten - 20-percent uranium dioxide material is considerably more creep resistant than unalloyed tungsten.

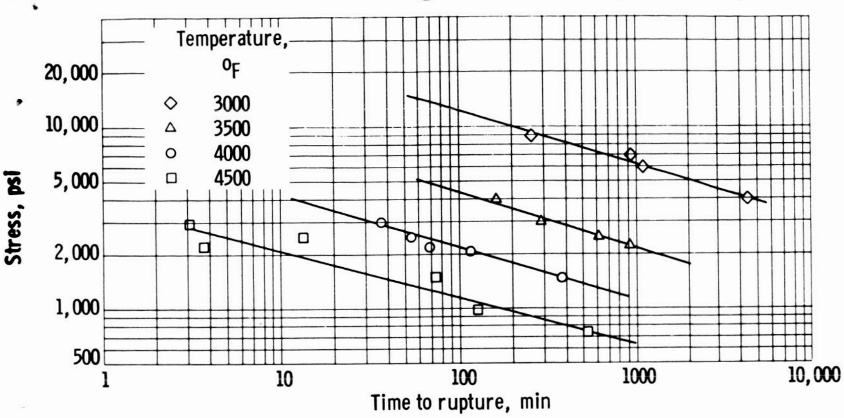

Figure 8. - Stress-rupture characteristics of tungsten - 20 percent uranium dioxide. Clad with 2 mil thick coating of tungsten.

The stress-rupture characteristics of the reference material are illustrated in figure 8. The allowable stress for rupture in 1 hour decreases from approximately 14,000 pounds per square inch at 3000° F to 1300 pounds per square inch at 4500° F. For a 10-hour rupture life, the allowable stress at 3000° F is 7000 pounds per square inch and at 4500° F is 700 pounds per square inch. These values are somewhat lower than those reported for unalloyed tungsten (refs. 11 and 12).

Metallographic examination of the creep specimens revealed that localized vaporization of the uranium dioxide occurred at 4500° F, though not necessarily at the fracture surface. From figure 9 it appears that grain boundary separation in the tungsten cladding occurred during creep, the uranium dioxide thus being exposed to the vacuum. As previously mentioned, uranium dioxide is known to vaporize quite readily in a high-temperature, low-pressure environment.

Figure 9. - Fuel loss through tungsten cladding during creep at 4500° F. Etchant, Murikami's reagent; X250. (Reduced 33 percent in printing.)

It is not known at present if the loss of uranium dioxide occurs at a relatively uniform rate during creep or, as seems more probable, if it is confined to the tertiary stage of creep. Further investigation in this area is necessary. No loss of uranium dioxide was noted in creep specimens tested at lower temperatures, although fracture was observed to be intergranular in all cases. Typical microstructures near the fractured surface of creep specimens are presented in figure 10.

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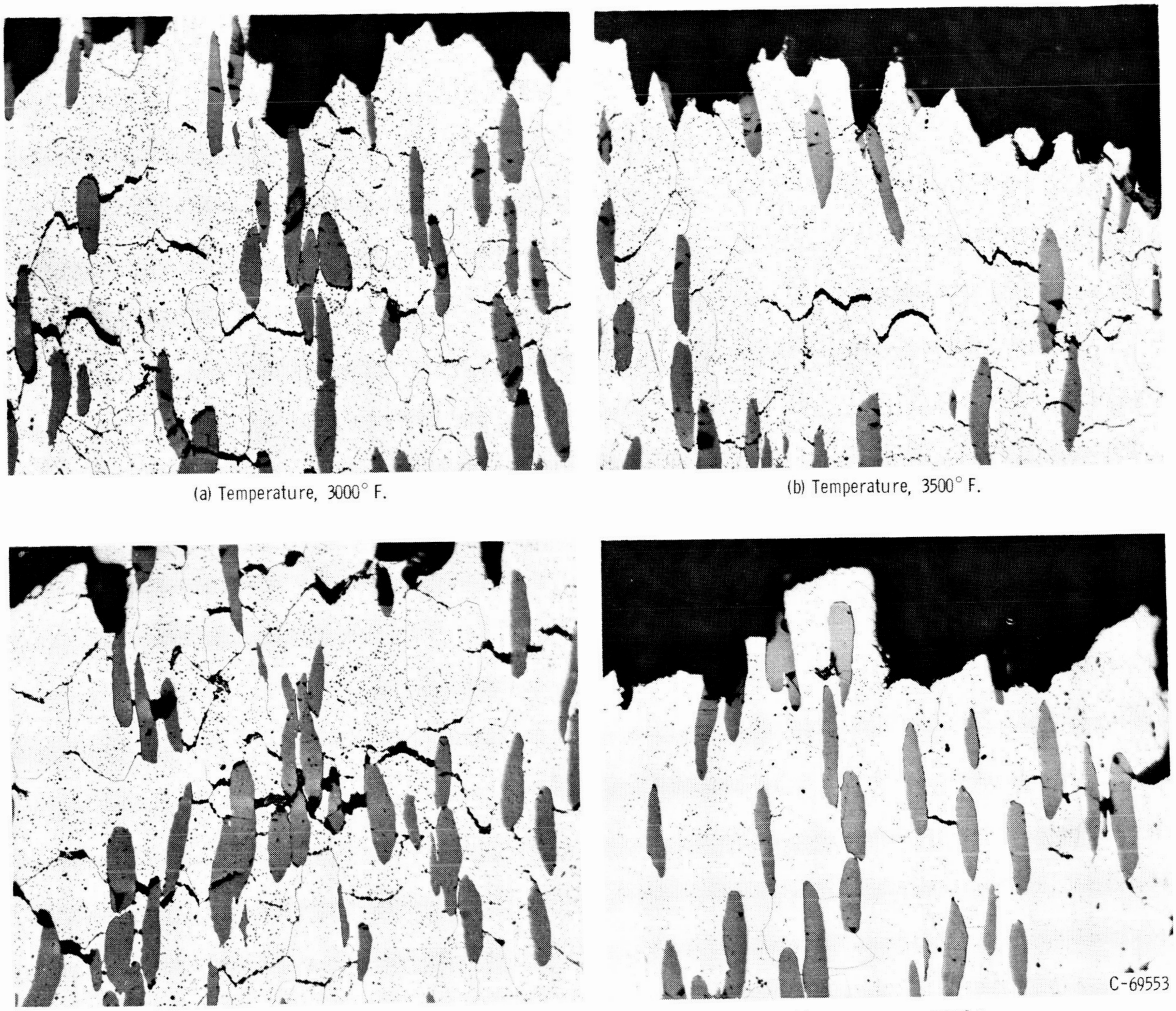

(a) Temperature, 3000° F.

(b) Temperature, 3500° F.

(c) Temperature, 4000° F.

(d) Temperature, 4500° F.

Figure 10. - Typical microstructures near fracture surface of creep-rupture specimens. Etchant, Murakami's reagent; X250. (Reduced 33 percent in printing.)

SUMMARY OF RESULTS

An investigation of the high-temperature mechanical properties of a tungsten-uranium dioxide composite yielded the following results:

1. The short-time tensile strength of tungsten - 20-percent uranium dioxide composites was slightly superior to that of similarly produced tungsten at elevated temperatures. The average short-time tensile strength of this material decreased from approximately 20,000 pounds per square inch at 3000° F to 1500 pounds per square inch at 5000° F.

2. There appeared to be no appreciable difference in tensile properties of tungsten - 20-percent uranium dioxide composites tested in either vacuum or hydrogen.

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3. The high-temperature tensile ductility of tungsten - 20-percent uranium dioxide composites generally decreased with increasing test temperature. The tensile ductility, as measured by reduction in area, varied from between 9 and 40 percent at 3000° F to between 8 and 16 percent at 4500° F.

4. The steady-state creep rate of tungsten - 20-percent uranium dioxide composites appears to be adequate for certain potential nuclear propulsion systems. At 4500° F, with a stress level sufficient to produce rupture in 1 hour, the creep rate was approximately 10^{-4} inch per inch per minute, while at stress levels low enough to permit a 10-hour rupture life, the creep rate was approximately 10^{-5} inch per inch per minute.

5. The stress-rupture properties of the tungsten - 20 percent uranium dioxide were somewhat less than those of unalloyed tungsten over the same temperature range. The allowable stress for rupture in 1 hour decreased from 14,000 pounds per square inch at 3000° F to 1300 pounds per square inch at 4500° F, while the stress for rupture in 10 hours decreased from 7000 pounds per square inch at 3000° F to 700 pounds per square inch at 4500° F.

6. At 4500° F, localized vaporization of the uranium dioxide through the tungsten cladding of the composite was observed. Since the vaporization occurred through separated grain boundaries in the tungsten cladding, it is more likely that the vaporization occurred during the tertiary stage of the creep rather than at a relatively uniform rate throughout the entire period of creep.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, June 5, 1964

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